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## Dislocation Dynamics in LiF and MgO Single Crystals

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Dislocation dynamics studies have been conducted in highly pure LiF and MgO single crystals using strain rate cycling and stress relaxation methods between 77 and 673 °K. In both materials, the values of the dislocation velocity-stress exponent  $m^*$ , obtained by these indirect methods do not compare well with  $m^*$  values determined using direct dislocation velocity measurements; in addition,  $m^*$  values obtained by indirect methods agree with each other only to a limited extent. Using the indirect methods at room temperature for example,  $m^*$  values between 3 and 16 are obtained in LiF, and values between 3 and 55 are obtained in MgO. The results indicate that the indirect methods for the determination of  $m^*$  are unreliable since its value depends on structural variables and the methods of measurement.

Es wurden versetzungsdynamische Untersuchungen von hochreinen LiF- und MgO-Einkristallen mittels Dehngeschwindigkeitsschwankung und Spannungs-Erholungsmethoden zwischen 77 und 673 °C durchgeführt. Die Werte des Versetzungsgeschwindigkeit-Spannungsexponenten  $m^*$ , die mit Hilfe dieser indirekter Methoden gefunden wurden, sind nicht in guter Übereinstimmung mit den Ergebnissen von  $m^*$ -Bestimmungen durch direkte Versetzungsgeschwindigkeitsmessungen; weiterhin stimmen  $m^*$ -Werte, die durch indirekte Methoden bestimmt werden, nur im begrenztem Umfang überein. Zum Beispiel wurden Raumtemperaturwerte von  $m^*$  zwischen 3 und 16 in LiF und zwischen 3 und 55 in MgO mittels indirekter Methoden gefunden. Die Ergebnisse deuten an, daß die indirekten Methoden für die  $m^*$ -Bestimmung auf Grund einer Abhängigkeit der Meßwerte und Meßmethoden und der strukturellen Variablen unzuverlässig sind.

### 1. Introduction

Plastic deformation in a crystalline material depends upon time, temperature, and strain rate and is a dynamic process involving the motion of dislocations. The imposed strain rate  $\dot{\epsilon}$  can be written as [1]

$$\dot{\epsilon} = b \rho_m \bar{v}, \quad (1)$$

where  $b$  is the Burgers vector of the dislocation,  $\rho_m$  is the mobile dislocation density, and  $\bar{v}$  is the average velocity of the mobile dislocations. The relationship between the average dislocation velocity and the effective stress applied on the dislocation line,  $\tau^*$ , is most often taken as a power law dependence of the form [2, 3]

$$\bar{v} = \left( \frac{\tau^*}{\tau_0^*} \right)^{m^*}, \quad (2)$$

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where  $m^*$  is the dislocation velocity-stress exponent in terms of effective stress and  $\tau_0^*$  is the effective stress required for unit dislocation velocity. Equation (2) has been found to be obeyed in LiF by Johnston and Gilman [4] and in MgO by Bruneau [5], both of whom studied dislocation velocity measurements using stress pulse techniques. It should be noted that the validity of (2) rests only on experimental grounds, and has no theoretical justification.

In the realm of dislocation dynamics, the dislocation velocity-stress exponent is generally considered to play an important role. This parameter is considered to represent the dynamic behavior of the material under an applied stress; for example, the smaller the value of  $m^*$ , the more pronounced will be the yield drop, or the delay time in creep tests.

Since the direct stress pulse method for determining  $m^*$  is difficult to carry out, a number of more convenient indirect methods have been proposed over the years for this determination. For many materials, however, these methods do not yield a unique value of  $m^*$ , and, in addition, discrepancies have been observed between values obtained by the indirect methods and those obtained by the direct method of dislocation velocity measurements, although few comparisons have been made on a systematic basis. In this paper, an evaluation of most of the available methods will be made with respect to  $m^*$ -determination in MgO and LiF.

## 2. Experimental Details

High-purity ultra-violet-grade LiF single crystals were obtained from the Harshaw Chemical Company cleaved to dimensions of  $10 \times 4 \times 4$  mm<sup>3</sup>. These crystals were annealed at 700 °C for 48 h and furnace-cooled to room temperature to standardize them and to anneal out any damage caused during cleaving. These LiF crystals contained less than 5 ppm of divalent cation impurities, and had an average critical resolved shear stress (CRSS) of 120 p/mm<sup>2</sup> at room temperature. For MgO, high-purity single crystals were obtained from the Oak Ridge National Laboratories. In order to get the greatest number of crystals from the same batch, these crystals were sawn into small-sized crystals with a diamond saw, using one (100) face as reference. The crystals were then cleaved to their final dimensions of approximately  $8 \times 2.5 \times 2.5$  mm<sup>3</sup>, following which they were annealed at 2000 °C for 2 h in a carbon element furnace with an argon atmosphere. This annealing treatment [6] ensured the presence of most of the iron impurity in the MgO as Fe<sup>2+</sup> and also served to standardize the specimens. These MgO crystals contained about 50 ppm Fe<sup>2+</sup>, and had an average CRSS of 1.1 kp/mm<sup>2</sup> at room temperature.

The LiF and MgO crystals were tested in compression using specially designed jigs [7] fastened to an Instron machine. Tests above room temperature were conducted using a nichrome-wound resistance furnace in one compression jig and tests below room temperature were conducted using another jig immersed into a low-temperature bath held in dewar flask. Strain-rate cycling experiments were done using the push-button cross head speed selector in the Instron machine at a particular temperature at various strains for strain-rate change ratios of 100, 20, 40, 10, 5, and 4. The change in shear stress,  $\Delta\tau$ , was determined from stress values before and after the change in strain rate; the temperature range for these tests was 77 to 673 °K. Double-strain-rate change experiments were performed using three strain rates such that [8]  $\dot{\epsilon}_2/\dot{\epsilon}_1 = \dot{\epsilon}_3/\dot{\epsilon}_2$ , at 198 and

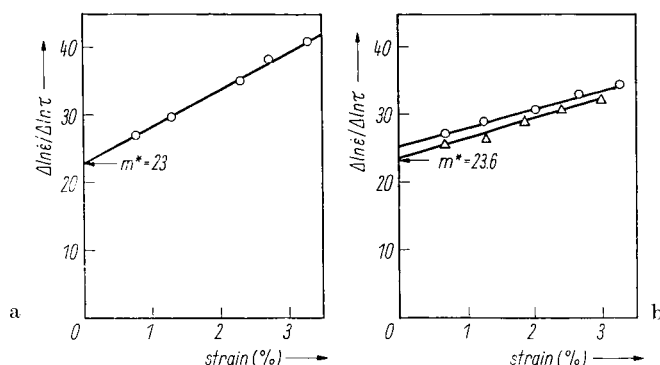


Fig. 1.  $m^*$ -determination by the extrapolation method in LiF single crystals. a) At 296 °K,  $\dot{\epsilon}_2/\dot{\epsilon}_1 = 20$ ; b) at 196 °K,  $\circ \dot{\epsilon}_2/\dot{\epsilon}_1 = 10$  and  $\triangle \dot{\epsilon}_2/\dot{\epsilon}_1 = 20$

296 °K for LiF and at 296 °K only for MgO. Stress relaxation tests were also performed at 77, 296, 473, and 523 °K for LiF and at 195 and 296 °K for MgO single crystals.

### 3. Results

#### 3.1 Extrapolation method

In a strain-rate cycling experiment,  $m^*$  can be obtained by extrapolating to zero plastic strain, the quantity  $m'$ , defined by [9]

$$m' = \left( \frac{\Delta \ln \dot{\epsilon}}{\Delta \ln \tau} \right)_e, \quad (3)$$

determined at a known strain. Fig. 1 shows the variation of  $m'$  with strain for LiF, obtained at 196 °K and at room temperature. Here,  $m'$  increases with strain and is dependent upon the strain ratio employed, increasing with decreas-

Table 1

Comparison of the dislocation velocity-stress exponent  $m^*$  in LiF obtained by various indirect methods at the lowest available strain. Values from the literature are shown for comparison

temper- ature (°K)	$m'$ (extra- polation)	Michalak's method	double- strain-rate change	stress relaxation	direct etch-pit technique
77	—	—	—	32	—
198	24	4.6	5.2	—	—
296	16	6.7	3.1	14	14 [4]
	15 [9]			6 [12]	7 [8]
					11.2 [16]
473	—	3.4	—	2.7	—
528	—	—	—	2.5	—
573	10	3.1	—	—	—

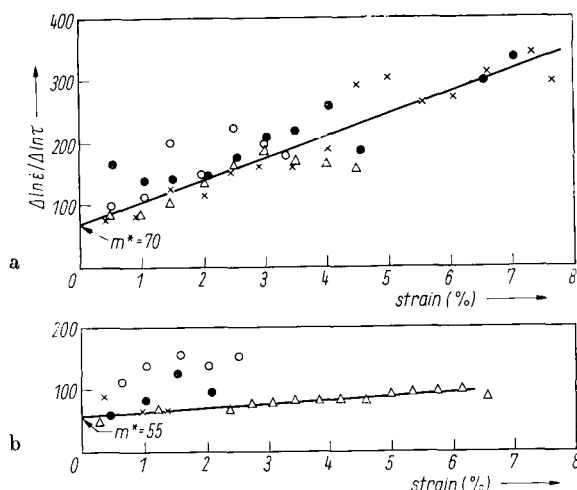


Fig. 2.  $m^*$ -determination by the extrapolation method in MgO single crystals. a) At 77 °K; b) at 295 °K;  $\dot{\epsilon}_1/\dot{\epsilon}_2 = 4$  (●), 10 (○), 20 (×), 40 (△)

ing strain-rate ratio at 196 °K. Overall, the observed value of  $m^*$  obtained using this method in LiF varies from 24 at 198 °K to 10 at 573 °K, as tabulated in Table 1.

In MgO there is, in general, a larger scatter in the data, and the value of  $m'$  at a given strain varies with the strain-rate ratio used, as illustrated at 77 °K and room temperature in Fig. 2. The  $m'$ -values increased with strain at 77 °K for all strain-rate change ratios, but are nearly constant with strain at 195 and 295 °K. Above room temperature,  $m'$  increases with strain. The  $m^*$ -value obtained by extrapolating the  $m'$ -values to zero strain vary from 70 at 77 °K to 32 at 673 °K, as given in Table 2.

Table 2

Comparison of the dislocation velocity-stress exponent  $m^*$  in MgO by different methods at the lowest available strains. Values from the literature are shown for comparison

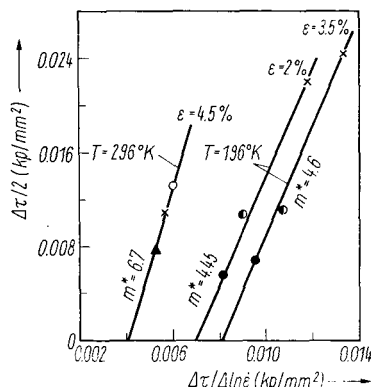
temper- ature (°K)	$m'$ (extrapolation)		Michalak's method	double- strain-rate change	stress relaxation	direct etch-pit technique
	current work	Phillip's data [8]				
77	70	29	1.8	—	—	—
195	52	15	8.6	—	—	—
295	55	11	3.2	4.5	39 2.1	4.6 [5]
473	46	9	—	—	—	—
673	32	8	—	—	—	—

### 3.2 Michalak's method

According to Michalak [10] and Li and Michalak [11], a plot of  $\Delta \tau / 2$  versus  $\Delta \tau / \Delta \ln \dot{\epsilon}$  obtained from strain-rate change data, will give a straight line of slope  $m^*$ , according to the relation

$$m^* \frac{\Delta \tau}{\Delta \ln \dot{\epsilon}} = \tau^* + \frac{\Delta \tau}{2}. \quad (4)$$

Fig. 3.  $m^*$ -determination using Michalak's method in a strain-rate cycling experiment on LiF single crystals.  $\dot{\epsilon}_2/\dot{\epsilon}_1 = 100$  ( $\circ$ ), 40 ( $\times$ ), 20 ( $\blacktriangle$ ), 10 ( $\bullet$ ), 4 ( $\bullet$ )



Here it is assumed that the internal stress and the mobile dislocation density remain unchanged during the strain-rate change. Fig. 3 shows such a plot at two temperatures for LiF; values obtained using this technique at other temperatures are given in Table 1. For the case of MgO, it is often the case that (4) does not yield a linear plot, and the subsequent  $m^*$ -values are somewhat uncertain, but the results are given in Table 2 for comparison with others in MgO. In both LiF and MgO, it may be noted that the values of  $m^*$  obtained at the lowest temperatures measured are lower than the values obtained at the next-highest temperature. This is an anomalous result, since  $m^*$ -values are expected to decrease with increasing temperatures. This anomaly has not been explained.

### 3.3 Double-strain-rate change method

Values of  $m^*$  have also been determined for LiF and MgO single crystals using the double-strain-rate change technique of Li [8]. Under the assumption that the mobility dislocation densities at the three strain rates used are such that  $\rho_{m2}/\rho_{m1} = \rho_{m3}/\rho_{m2}$ , and that the internal stress is constant for the strain-rate change, the internal stress  $\tau_\mu$  is given by [8]

$$\tau_\mu = \frac{\tau_2^2 - \tau_1 \tau_3}{2 \tau_2 - (\tau_1 + \tau_3)}, \quad (5)$$

where the subscripts refer to the part of the cycle involved. Making the further assumption that  $\rho_{m2} = \rho_{m1}$ , this yields

$$m^* = \frac{\Delta \ln \dot{\epsilon}}{\ln ((\tau_2 - \tau_\mu)/(\tau_1 - \tau_\mu))}. \quad (6)$$

Table 3 gives the values of  $m^*$  obtained as a function of strain in LiF and MgO using this double-strain-rate change test at 295 °K. In LiF, the  $m^*$ -values remain fairly constant with strain after an initial decrease, an observation similar to that of Gupta and Li [12]. In MgO, the  $m^*$ -values increase with strain. In all cases tested, the values of  $m^*$  obtained in LiF by double-strain-rate change tests are considerably smaller than the values obtained by the extrapolation method (see Tables 1 and 2).

Table 3  
The strain dependence of  $m^*$ , determined by double-strain-rate change tests at 296 °K

LiF		MgO	
strain (%)	$m^*$	strain (%)	$m^*$
2.70	3.1	0.33	4.5
2.71	3.0	0.86	3.6
4.46	2.2	1.17	4.0
4.98	2.6	2.01	7.8
8.38	2.3	3.72	9.4

### 3.4 Stress relaxation method

Stress relaxation tests in LiF indicate that in general the relaxed stress is a logarithmic function of relaxed time. Fig. 4 shows a typical set of data for LiF where  $m^*$  is obtained from the slope of the plot of  $d\tau/dt$  as a function of  $\lg t$ , according to Li's analysis [8]. The remainder of the determinations are given in Table 1. It should be noted that at 295 °K, the value obtained for  $m^*$ , 14, is somewhat higher than the value of 6 reported by Gupta and Li [12] using the same technique in similar crystals.

Stress relaxation curves for MgO generally show more scatter than those in LiF. The results are analysed using the methods of both Noble and Hull [13] and of Li [8]. According to Noble and Hull, a plot of  $\lg \tau$  versus  $\lg t$  should be linear, with a slope of  $(1 - m)^{-1}$ . Such analysis yields an  $m^*$ -value of 39 for MgO at room temperature. This is very high compared to the values obtained using Michalak's method and the direct dislocation velocity measurements [5], but is closer to the room temperature value of  $m^*$  obtained by the extrapolation  $m'$ . The use of Li's method for analysis of the stress relaxation curves gives a large amount of scatter from the expected linear behavior, as seen in Fig. 5. Li's analysis yields a value for  $m^*$  of 2.1, which conflicts with the above, but which is close to the value obtained from strain-rate change methods, as may be noted in Table 2. The cause for this discrepancy is not known.

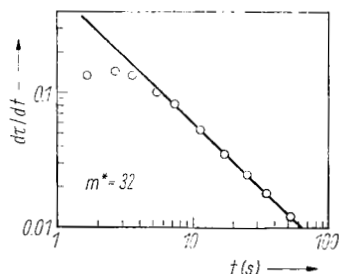


Fig. 4.  $m^*$ -determination from stress relaxation experiments, according to Li's analysis, in LiF single crystals at 77 °K. Prior strain: 2.0%

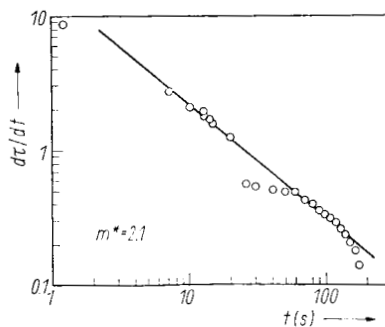


Fig. 5.  $m^*$ -determination from stress relaxation experiments, according to Li's analysis, in MgO single crystals at room temperature. Prior strain: 0.50%

#### 4. Discussion

In both LiF and MgO, there is a large scatter in the data leading to the determination of  $m^*$  by several of the methods employed, and in general the values of  $m^*$  obtained by the different indirect methods at different temperatures do not agree well with one another. In particular,  $m^*$  obtained by the extrapolation method is much higher than the values obtained by direct methods. Several reasons have been advanced for this. Li [8] has pointed out that the suggestion that this is due to an increase in the mobile dislocation density is not feasible although it is difficult to test the change in mobile dislocation density with a change in strain rate.

Another reason given for the discrepancy is the existence of internal stresses [2]. In both LiF [14] and MgO [15], the determination of  $\tau_\mu$  by different methods gives large values for the athermal component of the flow stress confirming in fact the existence of large internal stresses. The applied stress  $\tau_a$  is composed of the effective stress  $\tau^*$  and the internal stress  $\tau_\mu$  such that  $\tau_a = \tau^* + \tau_\mu$ . From this [10],

$$\tau_a \frac{\Delta \ln \dot{\epsilon}}{\Delta \tau} = \tau^* \frac{\Delta \ln \dot{\epsilon}}{\Delta \tau} + \tau_\mu \frac{\Delta \ln \dot{\epsilon}}{\Delta \tau}, \quad (7)$$

where the left-hand side of the equation is  $m'$  and the first term of the right-hand side of the equation is  $m^*$ . Therefore,

$$m' = m^* + \tau_\mu \frac{\Delta \ln \dot{\epsilon}}{\Delta \tau}. \quad (8)$$

Hence, large values of  $\tau_\mu$  will give large values of  $m'$  at a given strain, and extrapolating these artificially high  $m'$ -values to zero strain gives large values of  $m^*$ , as is evident from the present work.

Noble and Hull [13] have also pointed out that dislocations move through undeformed material during direct measurements of  $m^*$ , whereas when indirect methods are used,  $m^*$  is determined for the dislocations which move in already deformed material. Hence, back-stresses due to work-hardening are important for the indirect determinations, and the hypothetical situation of one dislocation moving through the crystal in the early stages of deformation, which is valid for direct methods, is no longer accurate. Hence equation (2) should be written [13]

$$\bar{v} = \left( \frac{\tau_f - \tau_b}{\tau_{f_0} - \tau_b} \right)^{m^*}, \quad (9)$$

where  $\tau_b$  is the effective back-stress due to work-hardening. The reason for the very high values of  $m^*$  obtained by the extrapolation method may therefore be due to the use of the externally applied stress, uncorrected for  $\tau_b$ , and the extrapolation of this artificially high values of  $m'$  to zero strain.

In strain-rate change tests, the underlying assumption inherent in the derivation is that the density of mobile dislocations, and the internal stress, remain unchanged during the change in strain rate. These assumptions are untested, particularly since it is difficult experimentally to measure changes in the mobile dislocation density. For LiF, there is good agreement between the values of  $m^*$

obtained from the extrapolation  $m'$  and from the stress relaxation tests, being 16 and 14 at room temperature, respectively. This is close to the value of 11.2 obtained by Soifer et al. [16] using pulse techniques in LiF single crystals containing 300 ppm Mg. However, the  $m^*$ -values obtained at 296 °K by Michalak's method and by double-strain-rate change tests, 6.7 and 3.1, respectively, are considerably lower. In addition, there is the anomaly of the lowest temperature values obtained using Michalak's method being lower than a higher temperature value, as noted earlier. Since  $\tau^*$ -values obtained by Michalak's method are comparable to those obtained by other methods [14], the assumption of the constancy of  $\tau_\mu$  is perhaps valid at this temperature.

For MgO, a large amount of scatter from the expected behavior is observed in stress relaxation experiments at both 195 and 295 °K. Also, stress relaxation gives different values of  $m^*$  when analysed in different ways. Since stress relaxation involves sufficient time in which dislocations may be able to rearrange themselves into low-energy configurations, it is difficult to assure constant dislocation substructure during relaxation. This could be the reason for the discrepancy of  $m^*$  in the stress relaxation method as compared to other methods. On the other hand, since it is more reasonable to assume that the dislocation density remains unchanged during an instantaneous change in strain rate [8], strain-rate change methods should be more reliable than stress relaxation in  $m^*$ -determinations, at least as far as MgO single crystals are concerned. It should be noted that even for stress relaxation tests conducted at room temperature where temperature fluctuations were nil, discrepancies existed.

In the strain-rate change method,  $m'$ -values increase with strain. This increase has been attributed to work-hardening. In the study of Thoronton et al. [17] on single and polycrystalline copper at room temperature,  $m'$  was found to be related to both structural changes and the mechanism of flow. For single crystals,  $m'$  was found to change from about 100 in the easy-glide region to about 225 in stage III deformation, where it became equal to the polycrystalline value. Hence the philosophy of the extrapolation method to obtain  $m^*$  can be questioned, since  $m^*$ -values should therefore be obtained by the extrapolation only for those data from a single work-hardening region. However, although both LiF [14, 18] and MgO [6, 15] show multi-stage work-hardening behavior, there is no systematic variation in  $m'$  found which depends upon the particular work-hardening stage encountered.

As has been demonstrated above, widely varying values of  $m^*$  are found using indirect methods in both LiF and MgO single crystals. As Nichols [19] has shown, the uncertainties in the variation of mobile dislocation density and of barrier shapes and spacings may lead to variations of the power law behavior on which the  $m^*$ -concept is based. Hence  $m^*$  could differ widely for different methods of determination. Bailey and Flanagan [20] have taken note of the dependence of the mobile dislocation density with strain-rate change, and have proposed that  $m^*$  is not an intrinsic material deformation parameter since it depends upon structural variables and methods of measurement. The present study shows that the indirect methods of determination of  $m^*$  do not yield a unique value in either MgO or LiF, indicating that they are unreliable in determining the deformation parameter for these crystals. This, combined with experimentally determined variations in  $m^*$  in other systems, and the fact that the power law relationship has no theoretical basis, indicates that  $m^*$ -determinations in general may have doubtful significance.



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